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DEPARTMENT OF COMMERCE

UNITED STATES BUREAU OF MINES
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INFORMATION CIRCULAR

TANTALUM

(AND COLUMBIUM)

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BY

E. P. YOUNGMAN

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DEPARTMENT OF COMMERCE--BUREAU OF MINES

TANTALUM[1]

(and Columbium)

By E. P. Youngman[2]

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[1] The Bureau of Mines will welcome the reprinting of this paper, provided the following footnote acknowledgment is used: "Reprinted from U. S. Bureau of Mines Information Circular 6328."

[2] Rare metals and nonmetals division, U. S. Bureau of Mines.

INTRODUCTION

Tantalum is a rare element; estimates indicate that there is less tantalum than gold in the earth's crust. After remaining a laboratory curiosity for many years, it was used to a limited extent and for relatively few years (1903-1911) for incandescent electric-light-bulb filaments. Another wave of interest in tantalum appeared when its electrolytic-valve action was taken advantage of in charging storage batteries, especially for radio work. Tantalum chargers were eventually displaced by other types of chargers, but for a time they were manufactured in sufficiently large numbers to create a demand for the metal and to bring about its production on a commercial scale in the United States. As tantalum became available in larger quantities and at substantially lower prices, and as high prices for platinum encouraged the employment of tantalum as a substitute, the metal was put to many new uses. Ferrotantalum was made and sold before the World War, and patents have been taken out for a number of special alloys for industrial purposes, but probably the largest use for the metal to-day is in vacuum tubes.

The rapid increase in production reflects the increasing use to which tantalum is being put.

DESCRIPTION AND PROPERTIES

Commercial tantalum has a purity of at least 99.5 per cent. It is a platinum-white metal when polished, though it often has a steel-blue color, owing probably to a thin film on the unpolished surface. As indicated by the periodic table of the elements, tantalum (in its general characteristics) has much the same relation to columbium, its sister metal, that tungsten has to molybdenum. Its atomic number is 73, and it stands in the periodic chart in Group 5, adjacent to tungsten in Group 6. Its tensile strength is two and one-half times that of hard platinum, and it can easily be made as hard as iridioplatinum.[3] When tantalum is used cold, it appears that platinum alone has properties that rival it. Next to tungsten, tantalum has the highest melting point and the lowest vapor pressure of all metallic elements.

A summary of the technical characteristics and the chemical properties of commercial tantalum as given by the Fansteel Products Co. (Inc.), of North Chicago, Ill., follows:

Technical Characteristics

Atomic number	73
Atomic weight	181.5
Density, at 20°C, ...	16.6
Atomic volume	10.9
Tensile strength, lbs. per sq. in.	130,000
Compressibility per unit volume per kg/cm²	$.52 \times 10^{-6}$
Brinell hardness at 500 kg.	45.9
Scleroscope hardness	10

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[3] Manufacturers Record, Twelfth Exposition of Chemical Industries, vol. 95, No. 20, May 16, 1929, pp. 51-52.

Technical Characteristics (Cont'd)

Young's modulus of elasticity, kg. per sq. mm.	19,000
Melting point, °C.	2850
Boiling point, °C.	>4100
Vapor pressure	---
Heat of vaporization, total cal. per gm.	---
Specific heat, cal. per gm. per °C. at 0°C.	.0365
Linear coeeficient of expansion per °C.	7.9×10^{-6}
Thermal cond. in cal. per cm. per cm^2 per sec. per degree at 18° C.	.130
Heat of combustion, cal. per gm.	827
Heat of combustion cal. per gm. atom to oxide	300,120
Temp. coeeficient of resistance at 20° C.	.0031
Electrical resistance microhm per c.c. at 20° annealed	15.5
Magnetic susceptibility	$+.93 \times 10^{-6}$
Electrochem. equiv. mg. per coulomb	.3762
Refractive index	2.05
Thermoelectric e.m.f. against copper microvolts per degree	4.1

Chemical Properties

The most characteristic chemical property of tantalum is its unusual resistance to chemical corrosion. It is not attacked by hydrochloric or nitric acids or by aqua regia, either hot or cold. Dilute sulphuric acid does not attack it at ordinary or more elevated temperatures, but boiling concentrated sulphuric acid does appear to have some action upon it. Tantalum also is attacked with difficulty by solutions of caustic alkalies. In fact, hydrofluoric acid seems to be the obly chemical agent that will attack it. In the case of very pure metal and very pure hydrofluoric acid, even this action is very slow; but a mixture of hydrofluoric and nitric acids dissolves the metal with avidity, forming tantalum fluoride in solution.

If tantalum is heated in the air, the surface becomes blue at a temperature of about 400° C., and at a somewhat higher temperature nearly black. Above a dull-red heat, the white oxide is produced, and the metal gradually burns. This metal combines with hydrogen, oxygen, or nitrogen very readily. It will take up 740 times its own volume of hydrogen, producing tantalum hydride, a very coasse-grained, brittle product.

Tantalum containing dissolved gases will be harder than the pure metal, and if the quantity of the gases is appreciable, the metal may even be brittle; therefore, all annealing or heating operations with tantalum must be carried out in a vacuum. Solutions of chlorine and the gas itself are without any action on the metal. Tantalum is said to be unaffected by any of the chemicals or antiseptics used in dentistry or surgery.

The Fansteel Products Co. has published a table of corrosion data as a guide in determining possible commercial applications. The basic data were worked out by a committee of the American Society for Testing Materials, under the chairmanship of Jerome Strauss, and they relate to wrought metal in the form of sheet, wire, or bars. These data are tabulated as follows:

Corroding agent		Resistance[1]
Sulphuric acid	Dilute	Excellent
Do.	Concentrated and below boiling point	Excellent
Nitric acid	Very dilute	Excellent
Do.	Concentrated	Excellent
Hydrochloric acid	Dilute	Excellent
Do.	Concentrated	Excellent
Aqua regia		Excellent
Hydrofluoric acid		Poor
Hydrofluoric and concentrated nitric mixed		Poor
Acetic acid	10 per cent solution	Excellent
Do.	Concentrated (Glacial)	Excellent
Formic acid	12 per cent solution	Excellent
Do.	50 per cent solution	Excellent
Oxalic acid	10 per cent solution	Excellent
Phosphoric acid	10 per cent solution	Excellent
Do.	85 per cent solution	Excellent
Carbolic acid	5 per cent solution	Excellent
Citric acid solution		Excellent
Tannic acid	15 per cent solution	Excellent
Sodium acetate	20 per cent solution	Excellent
Iodine solution	10 per cent solution	Excellent
Chlorine solution		Excellent
Sodium hydroxide	Dilute solution	Good
Do.	Hot concentrated	Poor
Potassium hydroxide	10 per cent solution	Excellent
Ammonium hydroxide		Excellent
Sea water		Excellent
Sea air		Excellent
Moist atmosphere		Excellent
Moist sulphurous atmosphere		Excellent
Mine waters		Excellent
Gases containing carbon monoxide		Excellent

[1] "Excellent" means resistance to corrosion of such a degree in laboratory and service tests that a long life of years is obtained; "good," of such a degree that commercially long life is obtained; and "poor," of such a degree that the material is useless.

Working Properties

Tantalum can be worked cold, drawn, hammered, machined, polished, hardened, rolled, and punched. The pure metal is rather easily worked. The forms in which the metal may be readily obtained include wire from .001 inch in diameter up to heavy bar, sheet from .002 inch in thickness to varying widths, and tubing of various sizes. For a complete discussion, the reader is directed to M. M. Austin' "Working Properties of Tantalum."[5]

USES

Probably the largest use of tantalum now is in connection with the manufacture of radio and power tubes. Increasing quantities are being used in the manufacture of certain alloys, in the manufacture of spinnerets employed in the making of rayon, in the lining of chemical equipment, and in jewelry. A more detailed account of these and other uses follows.

With respect to further extension of the use of the metal, the chief problem is to ascertain to what extent it can replace platinum in the laboratory and factory and for acid-resisting equipment. It has not been proved that tantalum will provide a cheaper substitute for platinum in these connections. For precision weights, however, it is recognized internationally as a substitute for platinum. In electrical apparatus (radio valves, electrolysis, rectifiers), tantalum offers possibly a better service. Although, among the noble metals platinum alone is a strong rival, tantalum is limited in usefulness chiefly by the fact that it can not be raised above the temperature of about 350° without absorbing gases and without undergoing deterioration, and the further fact that when it is used hot it must be in a high vacuum.[6]

Metallic-Filament Lamps

The employment of tantalum as a lamp filament (introduced by Von Bolton) extended over the period between 1903 and 1911. Although tantalum filaments were more efficient than the old-fashioned carbon filaments, they were not so efficient as tungsten; and as soon as a way was found to make a tungsten filament that would withstand vibration, the use of the more expensive and rarer metal was practically discontinued, and tantalum was finally wholly replaced by tungsten.

Electrolytic Rectifiers

The second extended use made of tantalum was in electrolytic rectifiers (battery chargers). In 1922, the Fansteel Company put the rectifiers on a regular commercial basis because of the need of some means of charging storage batteries in radio work from an alternating current supply; and although other devices caused the consumption of tantalum for this purpose to decline, the chargers are now being sought for small storage batteries for cars, railway signals, medical apparatus,and like appliances.[7]

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[5] Austin, M. M., Working Properties of Tantalum; Am. Inst. Min and Met. Eng., Tech. Pub. No. 278, 1930, p. 8.

[6] Jones, Chester H., Tantalum -- A Metal for Difficult Corrosion Problems: Chem. and Met. Eng., Sept., 1929, p. 551.

[7] Holman, Tantalum: Min. Jour., vol. 168, No. 4927, Jan. 25, 1930, p. 10.

Tantalum has a very pronounced valve action. The usual type of tantalum rectifier consists of a lead and a tantalum electrode immersed in a suitable electrolyte and connected to input and output through a choke. "If two electrodes of tantalum are placed in sulphuric acid of accumulator strength, only 1 milliampere will pass when the potential across the electrodes is maintained at 75 volts."8/ The battery charger made by the Fansteel Products Co. is termed "Balkite"; the electrode used in chemically pure.

Laboratory Apparatus

In 1927, after tantalum became available in the United States at as low a price as 16 cents a gram (for sheet metal), Bishop & Co. (Malvern, Pa.) began the manufacture of chemical equipment, especially laboratory ware, including dishes, cathodes, and spatulas. The market for this purpose is now fairly steady.

The special value of tantalum for laboratory work and for lining chemical equipment is its resistance to corrosion. For these purposes its utility is limited, however, by the fact that it readily oxidizes in air when heated oven to temperatures below red heat. Its mechanical strength is another factor, its tensile strength being 130,000 pounds to the square inch in wire form. Its texture is more that of a closely woven wood fiber than that of the grain structures of the common metals.

Tantalum is not suitable, however, for all kinds of laboratory work for high-temperature ignitions (including alkali fusions) nor for many industrial chemical operations that are conducted at temperatures above red heat.

Electrolysis

Tantalum has great possibilities in connection with electrolysis on a large scale because it is not attacked by fused sodium chloride, and in the small-scale electrolysis of the laboratory it is superior to platinum for the qualitative determination of metals by electrolytic deposition.9/

Vacuum Tubes

One of the largest of the present uses of tantalum metal (that in connection with the elements of vacuum tubes employed in radio receiving and broadcasting, in X-ray and similar work, and in Neon lights) is owing to the fact that tantalum will absorb all the common gases when it is heated to a red heat, thus functioning as its own "getter", and will automatically "harden any vacuum in which it is heated, which fact, it is said, will account for an increase in the useful life of a valve."10/

The following is excerpted from an article by F. L. Hunter,11/ who discussed tantalum as a plate material, tantalum as a grid material, and tantalum in the field of gas-filled discharge tubes. These remarks, though applying to both receiving and power tubes, more particularly concern power tubes.

8/ Dyson, G. Malcolm, The Production and Uses of Tantalum: Chem.Age, Mar.2,1929. p. 78.

9/ Dyson, G. Malcolm: Work cited, p. 19.

10/Dyson, G. Malcolm: Work cited.

11/Hunter, F. L., Tantalum in Vacuum Tubes: Radio Eng., vol. 9, No.12, Dec., 1929, pp. 20-31.

1. Tantalum more than any other material fills the following requirements of plates for air-cooled power tubes:

(1) Ductility and workability.
(2) Ability to stand high temperature.
(3) Complete and permanent degassing.
(4) The ability to form joints with the same or different material by riveting or welding, which retain their strength after prolonged heating at high temperatures.

2. Accuracy of grid dimensions and resistance to deformation throughout a wide range of temperatures determine the uniformity of tube characteristics. The springy, semiflexible structure must take a permanent set to definite dimensions with an accuracy of plus or minus .001 inch and retain that set through handling, mounting, and bombarding. Tantalum alone possesses the physical and chemical properties that grid wires in the completed tube should have, which are as follows:

(1) High tensile strength combined with moderate and constant ductility.
(2) Low initial gas content with rapid and complete evolution of occluded gases at low temperatures (600 to 700° C.).
(3) The grid wires must be chemically inert toward thermionically active material thrown off the filament and toward condensed flash material.

Spinnerets

Recently tantalum has been used successfully in the manufacture of spinnerets for the rayon industry. Not only does it resist corrosion, but since it can be coated with an electrolytic oxide of almost diamond hardness, there is no apparent wear or distortion of the holes through which the spinning solution is ejected. Like tantalum, the oxide coating is inert toward all chemical reagents except hot fused caustic, hydrofluoric acid, mixtures of hydrofluoric and nitric acids, and 95 per cent boiling sulphuric acid.[12] Despite these advantages, the use of tantalum is by no means universal in the manufacture of rayon. According to Carter, [13] "some platinum cups are used, but more generally gold, containing up to 30 per cent platinum or palladium, is the preferred metal."

Miscellaneous

Calorimeters.—In 1927 or 1928, the Pacific Experiment Station (Berkeley, Calif.), of the Bureau of Mines, constructed a new type of precision calorimeter for studying the dissolution of metals and oxides in strong acids. Part of the calorimeter is a cylindrical can with hemispherical bottom and flanged top, which must withstand the action of strong mineral acids over long periods. In experiments, 2.5 normal hydrochloric acid and acidulated copper solutions have been used.

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[12] Jones, Chester H., Work cited, p. 551.

[13] Carter, Frederic E., Precious Metals for Ultimate Corrosion Resistance: Chem. and Met. Eng., vol. 36, No. 9, Sept., 1929, pp. 553-555.

The mechanical usage is severe, since the contents of the container are constantly stirred, and at one step of the experiments glass containers are smashed directly upon the hemispherical bottom. A tantalum and bakelite construction has proved very satisfactory, not only from the standpoint of corrosion resistance and mechanical strength and toughness, but from the standpoint of economy. Containers made of platinum or gold (materials formerly used) cost over $1,500 and $300, respectively, whereas a container made of tantalum costs $150. If gold were used, difficult reinforcing problems would arise, because fine gold would have to be employed.

Jewelry.—The use of tantalum in jewelry has been reported by the press and by the Fansteel Products Co., not as a substitute for platinum, but as a new element in design. Holman says,[14] "Electrolytically, tantalum can be coated with a film of tantalum oxide. Through adjustment of the thickness of this film, various brilliant colors, actually interference colors, can be produced on the surface of the metal. These colors are stated to be durable and to resist wear." Beads, wrist-watch cases, and straps are listed as the articles in which tantalum is employed.

Pen nibs.— The use of tantalum in pen points was patented by Siemens & Halske[15] in 1905. An alloy[16] of 90 per cent tantalum and 10 per cent tungsten has been employed for this purpose.

Spectacle frames—"Add spectacle frames to its many uses—it can be spot-welded," reads an advertisement of the Fansteel Co.

Pipe lining or covering.—Tantalum is now successfully used as the lining or the covering for pipes, since it can be spot-welded to iron or steel.

Tungsten-carbide tools.—Tantalum has been proposed as a bonding agent for tungsten carbide in material of the "carbaloy" type, replacing cobalt.

Alloys

Many alloys of tantalum have been described, though few have entered industry. The alloys may be conveniently grouped in three classes, as follows:

Alloys of high melting point.—The alloys of high melting point, containing from 1 to 40 per cent tantalum, are combined with different proportions of molybdenum, tungsten, carbon, manganese, nickel, vanadium, and silicon. Tantalum, it is claimed, may replace all or part of the tungsten or the molybdenum.

Hard alloys.—The hard alloys, containing as much as 50 per cent of tantalum, combined with iron, chromium, vanadium, tungsten, molybdenum, or carbon, are employed in the manufacture of tools. Ferrotantalum-columbium (niobium) has been produced for many years by a Liverpool firm for use especially in high-speed steel.

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[14] Holman, ____, Tantalum: Min. Jour., vol. 168, London, Jan. 25, 1930, p. 10.

[15] Siemens & Halske A.-G., Manufacture of Writing Pens of Metallic Tantalum and Alloys: British Patent 3691-B, 1905.

[16] Engle, _____, and Austin, _____, Tantalum in Pens: Reviews and Abstracts of Tech. Periodicals; U. S. Patent, 1,701,399: Foote-Prints, vol. 2, No. 1, 1929, p. 31.

Probably more than 100 tons of this alloy have been produced and sold since manufacture was started.

Acid-resisting alloys.--The amount of tantalum used in acid-resisting alloys seems to be rather small. Since the introduction of the first rustless steel (an alloy of tantalum and iron), other alloys have been developed that have caused keen competition. Recently alloys of tantalum with iron and aluminum have come into existence.[17]

HISTORY

In 1801 Hatchett discovered the oxide of a new element while he was examining a black mineral from Haddam, Conn., which had been presented to the British museum. He named the mineral "columbite" and the metal "columbium." A year later a Swedish investigator, Eckeberg, while working with a new mineral, yttrotantalite, discovered a new element, to which he gave the name of tantalum (for Tantalus, of Greek mythology) because "when placed in the midst of acids it is incapable of taking any of them up and saturating itself with them." In 1809 Wollaston attempte to show experimentally that columbium and tantalum were identical, but it was soon proved that they were two separate elements. Subsequently, a number of investigators found other "new elements" in tantalum-and columbium-bearing ores, but these were shown to be mixtures of the twin metals. In 1866 Marginac completed his method for their separation, which was based upon the difference in solubility of their double fluorides with potassium.

The first mention of tantalum in metallic form is in connection with Berzelius, who in 1824 obtainted an impure product by reducing potassium-tantalum fluoride with potassium. This product contained not more than 60 per cent of metal and had a specific gravity of 10. In 1902 Moissan produced a very hard and brittle form of tantalum that was high in carbon and had a specific gravity of approximately 12.8. In 1903, in Germany, Dr. W. von Bolton evolved a process for the production of tantalum of a purity to enable drawn filament wire to be produce from it, from which incandescent lamps were made. Between 1905 and 1911, probably more than a million of these lamps were manufactured. The materials as a filament was then replaced by tungsten, and tantalum was almost forgotten for 20 years, when its special electrolytic-valve action was made use of. Tantalum was first refined as a pure worable metal in commercial form by Dr. C. W. Balke, of the Fansteel Products Co. (Inc.), of Chicago.

MINERALS

Tantalum and columbium (or niobium), almost invariably found together, occur in a large variety of minerals, none of which, however, are very widely distributed. Tantalite, skogbolite and tapiolite (tantalates of iron and manganese), and microlite (a calcium tantalate) are the richest in tantalum. Tantalite, or columbo-tantalite, is the principal commercial source of the metal.

Pure tantalite is a tantalate of iron, $Fe(TaO_3)_2$; pure columbite is a columbate of iron, $Fe(CbO_3)_2$. The two minerals form an isomorphous series-- part of the columbium in columbite almost always being replaced by tantalum, and part of the tantalum in tantalite almost always being replaced by columbium. There is almost complete gradation of one mineral into the other; neither is found pure. In some varieties, manganocolumbite or manganotantalite, the iron is largely replaced by manganese. Often a part of the iron is replaced by small amounts of titanium, tin, and tungsten. The columbite of the Black Hills contains from 18 to 40 per cent of tantalum oxide (Ta_2O_5); but in Australia, ore containing 70 and even 75 per cent or more tantalum oxide is mined.

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[17] Sears, George W., Progress in the Production and Use of Tantalum: Am. Inst. Min. and Met. Eng. Tech. Pub. No. 279, 1930, p. 4.

The principal other minerals in which tantalum occurs are pyrochlore (which differs from columbite in that calcium replaces the iron and that rather large quantities of thorium and cerium are present); rare-earth minerals, such as yttrotantalite, fergusonite (best variety in Western Australia, with 54 parts of tantalum oxide to about 2 parts of columbium oxide), and euxenite; and a comparatively recently discovered mineral,18/ strüverite, of which there are extensive deposits in Malaya, at Sungei Sebantun, and which is a titaniferous-tantalum ore. (This Malay deposit, which is thought to be a potential source of large quantities of both tantalum and titanium, is a kaolinised granite intrusion in hardened black shale.)19/ Samarskite is a columbo-tantalate, of which about one-third the mineral acid is that of tantalum. It is found in Virginia, Colorado, and especially at Mysore, India, where pieces weighing several pounds have been found.20/

IDENTIFICATION

Tantalite is black and has a dull metallic luster and a high specific gravity; it has poor cleavage and a hardness a little too great to permit scratching with a knife. Field identification of tantalite or columbite is difficult; only by quantitative chemical analysis can they be accurately differentiated, and a qualitative chemical test of some sort is usually necessary even to avoid confusing the colombo-tantalite mixtures with ilmenite, magnetite, cassiterite, tourmaline, and wolframite. Ilmenite may be differentiated by a test for titanium. Magnetite, often occuring in the same association, may be distinguished by its octahedral cleavage and by its magnetic quality. Cassiterite is often yellowish brown and translucent on thin edges, whereas tantalite is always black and opaque; the crystals of cassiterite are almost equidimensional, and those of tantalite are flattened and tabular; the color of the fine powder of cassiterite is gray or light brown, and that of tantalite is dark brown to black. Cassiterite may be reduced to metal by being placed in hydrochloric acid with zinc. Tourmaline may be distinguished by being weighed in the hand, as the spcific gravity of tantalite is much higher. Wilframite has a good cleavage and a shining metallic luster on the cleavage face, whereas tantalite fractures irregularly and has a dull fracture surface.

In order to make a definite test for tantalum,21/ separate the mixed tantalic and niobic oxides by fusion with potassium bisulphate, as follows:

18/ Hess, Frank L., and Wells, Roger C., An Occurrence of Strüverite: Am. Jour. Sci. vol. 31, May, 1911, pp. 432-442.

19/ Dyson, G. Malcolm, Work cited, p. 78.

20/ James, C., Tantalum: Mineral Foote-Notes, Aug., 1917, p. 1.

21/ Brush, George J., Manual of Determinative Mineralogy, John Wiley & Sons, New York City, 1901, pp. 123-124 and p. 99.

The fusion (of niobates and tantalates with 8 to 10 parts of potassium bisulphate) is ordinarily done in a crucible, but it may also be made in a test tube, since the heat which is required need not exceed faint redness and the glass is not attacked. When the decomposition is complete, as shown by the disappearance of dark particles, the tube may be inclined and turned while cooling, causing the fusion to solidify as a thin crust on its sides, so that it may be more rapidly dissolved on subsequent treatment. It is digested with cold water, which requires considerable time (the application of heat is not recommended), and there is left an insoluble white residue consisting of niobic and tantalic oxides, while the bases are in solution. The insoluble oxides are collected on a filter and washed, and if a portion of them is treated in a test tube with hot concentrated hydrochloric acid and boiled with granulated tin, the blue color due to niobium may be obtained.

And then in order to confirm the presence of tantalum--

Treat the oxides in a platinum dish with a little pure hydrofluoric acid (filtering if necessary) and add a little potassium fluoride. Evaporate the solution in a water bath nearly, but not quite, to dryness; dissolve the residue in the smallest possible quantity of boiling water; and allow the solution to become cold, when, if, tantalum is present, a very characteristic double salt (K_2TaF) crystallizes out in fine needles. The crystals, if collected on a filter paper and dried, have the appearance of wool. It is necessary that the hydrofluoric acid should be free from hydrofluosilicic acid (alone it should give no precipitate with potassium fluoride) and platinum or silver vessels must be used.

Powell and Schoeller,[22] who discuss the inadequacy of the usual qualitative tests for tantalum, advocate the tannin method for the separation of tantalum from niobium (columbium), which is shown to be applicable to the detection, as well as the determination, of small quantities of tantalum in niobium compounds and to the certain identification of the two elements in admixture by the production of precipitates of characteristic color. This method is "based upon differential hydrolytic dissociation between oxalotantalic and oxaloniobic acids in presence of tannin in slightly acid solution. The method possesses the following advantages: The color of the tantalum precipitate indicates at a glance whether it is free from, or contaminated with, niobium; no platinum vessels are required; and the accuracy approximates that reached in the separation of most of the common elements."

Early in 1929, the Rare and Precious Metals Experiment Station of the Bureau of Mines, in cooperation with the University of Nevada, made use of a new method for the separation of these two elements, which is quicker and cheaper. The method, of which the following paragraph is a summary, was published by George W. Sears, consulting chemist of the Reno station:[23]

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[22] Powell, Alan R., and Schoeller, Walter R., Investigations into the Analytical Chemistry of Tantalum, Niobium, and their Mineral Associates; A New Method for the Separation of Tantalum from Niobium; The Detection and Determination of Tantalum in Niobium Compounds: The Analyst, vol. 50, 1925, pp. 485-498.

[23] Sears, G. W., Critical Studies of the Fusion of Rare Metal Ores; III, Determination of Tantalum and Columbium: Jour. Am. Chem. Soc., vol. 51, No. 1, Jan. 1929, p. 128.

The procedure (finally adopted as giving the most satisfactory results) consisted in fusing the weighed sample at about 650° with 12 to 13 parts of sodium pyrosulphate and leaching the cold melt with water to remove it from the crucible and dissolve most of the excess sodium sulphate. The clear supernatant liquid is then decanted through a filter, the residue digested with hot 3 N hydrochloric acid to remove iron and titanium, transferred to the filter, washed free from sulphate with hot water, dried, and ignited. This product contains all of the tantalum and columbium, along with a small amount of silica, which is removed by treatment with hydrofluoric and sulphuric acids. After weighing the pure mixed oxides, a definite portion is transferred to a porcelain crucible and fused in the furnace with 5 grams of sodium pyrosulphate. The temperature is slowly raised to 850° and held at 835 to 850° for 10 to 15 minutes. It is then removed from the furnace, leached with water, filtered through a Gooch crucible, provided with an asbestos pad, and washed two or three times with hot 3 N hydrochloric acid. The residue, together with the asbestos pad, is then transferred to the original beaker, with the aid of as little water as possible, and 15 centimeters of concentrated sulphuric acid is added. The mixture is then evaporated to the appearance of white fumes and kept at that temperature for 10 to 15 minutes with occasional stirring. When cold the concentrated acid solution is slowly poured with constant stirring into 100 centimeters of water kept cold with ice or running water. The mixture is then filtered, washed with cold water, and the columbic acid precipitated from the filtrate with ammonia. The precipitate is filtered, dried, ignited, and the silicia removed by treatment with hydrofluoric acid and sulphuric acid. The final residue is weighed as columbium oxide (Cb_2O_5).

EXTRACTION

Since at the present time columbium, having no commercial importance, only increases the cost of tantalum, a grade of tantalite containing at least 60 per cent of tantalum oxide and only a few per cent of columbium is generally used.

Hand-picked, crushed ore is ground to a fine powder and then fused with twice its weight of acid potassium sulphate. The fused mass, while hot, is poured into large earthenware vessels full of water. Disintegration of the melt is completed by agitation with live steam. Most of the impurities dissolve during this process, the residue being an impure mixture of tantalic and columbic acids, which are separated by sedimentation and treated with ammonium-sulphide solution, to remove tin and tungsten; and the resulting residue from the latter process is boiled with dilute hydrochloric acid, in order to remove any iron sulphide that may be present. Hydrofluoric acid is employed to separate the crude mixture of tantalic and columbic acids. Two methods of conducting this separation are as follows:

1. The crude acids are dissolved in strong hydrofluoric acid of such a strength that the solution contains 14 per cent of tantalum oxide. This solution is raised to the boiling point, and an excess of potassium fluoride is added. On cooling, potassium tantalofluoride separates into small crystals. A variant of this process is to fuse the crude acids with acid ammonium fluoride and precipitate as before with potassium fluoride. There is always the possibility that some potassium titanofluoride will be precipitated along with the corresponding tantalum salt, but it may be removed almost entirely by washing with water. (7922)

2. The crude oxides are fused with potassium hydroxide, in order to obtain the soluble tantalates and columbates, which are leached with water and converted into the purified acids by precipitation with sulphuric acid. The purified precipitate readily dissolves in hydrofluoric acid, and on the addition of potassium fluoride the compound K_2TaF_7 is precipitated. The columbium is converted to $K_2CbOF_5.H_2O$, which remains in solution, being twelve times as soluble as potassium tantalofluoride. If the metal is the ultimate product aimed at, the tantalofluoride is boiled with ammonia, and the acid is filtered off and ignited, when tantalum oxide is obtained in a state of comparative purity.[24]

Simpler methods of extraction would bring about a greater use of tantalum and would proably introduce columbium to industry. The work that is in progress to apply on a larger scale the principle for the quantitative determination of the two elements (see p. 12) that has been developed in the laboratory of the University of Nevada has not gone far enough for a prediction to be made as to its success. It is not to be hoped that tantalum and columbium will ever be separated as easily as are the other rare metals, because of the similarity in the chemical properties, the acidic nature of their compounds, their great inactivity toward common reagents, and their strong tendency to remain in chemical combination.[25]

WORLD PRODUCTION

Tantalum minerals, though known to exist in many mineralized localities throughout the world, are produced in commercial quantities in but a very few—Western Australia ranking first, followed by South Dakota (United States). There is enough interest in tantalum in Northern Australia, in Rhodesia, and in Southwest Africa (a mandate of the Union of South Africa) to justify the expectation that these countries will soon produce small quantities at least, and a property in Northern Australia (recently optioned) is reported to contain one of the largest known deposits of tantalite in the world.

The Fansteel Products Co. (North Chicago), which in 1922 began the production of metal in sheets and rods large enough to be employed in pen points, dental instruments, chemical apparatus, and like equipment, is the only firm in the United States that is manufacturing the metal. A platinum company (Bishop & Co.) at Malvern, Pa., purchases the metal from the Fansteel Company for the manufacture of chemical apparatus and quipment.

Abroad, the principal purchaser of ore is George C. Blackwell Sons & Co. (Ltd.), of Liverpool (England); this company also manufactures a tantalum-niobium alloy. Guthrie & Co. (Ltd.), Accrington (England), and Siemens & Halske A.-G., Charlottenburg (Germany), are manufacturers of apparatus in ductile tantalum.

DOMESTIC PRODUCTION AND DEPOSITS

A little columbite was produced in the United States as early as 1904 and shipped abroad; there was a smaller production in 1905; in 1906 a commercial production was reported from Colorado and South Dakota, which went to Germany, and a few pounds were reported from North Carolina; in 1907 a small production of unknown weight and value, probably from South Dakota and North Carolina, was shipped to France; in 1908 no output was reported; in 1909, 1910, and 1911 small quantities were reported as mined but not as marketed; and from 1912 to 1918 there was no production reported. The following table gives production from 1918 to 1929.

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[24] Dyson, G. Malcolm, Work cited, pp. 18-19.
[25] Sears, George W., Work cited, pp. 5-6.

Columbite, mined in the United States, shipped in 1918-1929.

Year	Pounds	Value	Year	Pounds	Value
1918	4,500	$2,250	1924	1,197	$598
1919	300	90	1925	-	-
1920	4,000	1,450	1926	2,100	650
1921	3,400	1,150	1927	1,100	378
1922	600	240	1928	34,899	26,332
1923	1,350	540	1929	22,117	17,261

South Dakota

Since 1918, all of the tantalum reported has come from the Black Hills of South Dakota. In 1929, as in recent years, it came from the mines of the Black Hills Tin Co. (at Tinton), of Collingwood & Greene (near Pringle), and of A. T. Roos, Dakota Feldspar Co. (near Rapid City). The quantity shipped in 1929 amounted to 22,117 pounds, valued at $17,261, against 34,899 pounds in 1928, valued at $26,332, and 1,100 pounds in 1927, valued at $378.

As far as the records of the Bureau of Mines show, the Black Hills Tin Co. was the first in this country (1928) to mill and concentrate its own ore. Most of the ore in 1928 was sold to England and Germany. The material shipped carried 75 per cent or more of the combined pentoxides of tantalum and columbium, of which 45 per cent or more was tantalum pentoxide (Ta_2O_5).[26]

In South Dakota (the Black Hills), the principal source of tantalum is columbite, which occurs in pegmatite dikes where it is closely associated "with the minerals spodumene, beryl, albite (clevelandite), and muscovite. A common occurrence at the Ingersoll mine is in flattened, tabular crystals embedded in mixtures of quartz, albite, and muscovite. Crystals vary greatly in size. A crystal in the museum at the School of Mines weighs 33 pounds. Masses up to 600 pounds in weight have been reported from the Etta mine, and one large mass found at the mine is said to have weighed 2,000 pounds."[27] In the Tinton district of the northern Black Hills, tantalite concentrates are being produced. The Beecher lode near Pringle is also producing high-grade tantalite. The Hugo,

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[26] Hess, Frank L., Rare Metals: Mineral Resources of the United States, 1928, Pt. I, Bureau of Mines, 1929, p. 115.

[27] Connolly, Joseph P., and O'Hara, Cleophas C., The Mineral Wealth of the Black Hills: S. Dak. School of Mines, Bull. 16, May, 1929, p. 256.

Peerless, and other smaller mines yield columbite. Strüverite [28] is found in some quantity in the pegmatite dike on which the Etta claim is located, 1½ miles south of Keystone. For a complete geological discussion of the South Dakota deposits, see United States Geological Survey Bulletin 330.[29]

Other Localities

Whereas the deposits of tantalum minerals in South Dakota are the only commercially important sources of tantalum metal in the United States, columbite is a very common associate mineral of the pegmatite dikes of the eastern United States. The United States Geological Survey[30] reports columbite in California, Colorado, Connecticut, Maine, North Carolina, South Dakota, and Virginia; fergusonite in Virginia and Texas; samarskite in North Carolina; and tantalite in Virginia and South Dakota. Real tantalite has been reported in Georgia, also. Hatchettolite occurs in North Carolina, sparingly associated with samarskite at a number of mica mines in Mitchell County, N. C. (principally the Wiseman mine). Microlite has been found in octahedral crystals at Chesterfield, Hamshire County, Mass.; Branchville, Fairfield County, Conn.; and Amelia, Amelia County, Va.[31]

A promising locality for obtaining columbite in quantity is near Branchville, Conn.,[32] where the mineral occurs in some abundance in a pegmatite dike, associated with spodumene, in large masses and fine crystals. When this deposit was being worked for spodumene and feldspar, many barrels of columbite were shipped to universities for experimental work. Near Glastonburg, Hartford County, Conn., columbite was found in a feldspar quarry, but no attempt was made to use it.[33] It was to a specimen of ore from Haddam, Middlesex County, Conn., that the [illegible] name "columbium" was first given.[34] Hatchett, while studying minerals in the British Museum in 1801, was attracted by the rather high specific gravity and the bright black color of the specimen and began to experiment with the new element. (See previous section entitled "History".) The Cape Ann masses of alkali granite contain irregular patches and lenses of pegmatite, from which 32 minerals have been recorded. These include tantalite with danalite, cryophyllite, fayalite, gadolinit and others.[35] For further description, see the footnote reference below.[36]

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[28] Hess, Frank L., and Wells, Roger C., An Occurrence of Strüverite: Am. Jour. of Science, vol. 31, May, 1911, p. 433.

[29] Hess, Frank L., Rare Metals: U. S. Geol. Survey Bull. 380, 1909, pp. 131-133.

[30] Schrader, Frank C., Stone, Ralph W., and Sanford, Samuel, Useful Minerals of th United States: U. S. Geol. Survey Bull. 624, 1917, pp. 367, 372, 397, and 404.

[31] Pratt, Joseph Hyde, Production of Monazite, Jircon, Gadolinite, and Columbite or Tantalum Minerals: Mineral Resources of the United States, 1904, U. S. Geological Survey, 1905, p. 1226.

[32] Pratt, Joseph Hyde, Work cited, p. 1225.

[33] Pratt, Joseph Hyde, Work cited, p. 1316.

[34] Smith, Edgar F., Observations on Columbium and Tantalum: Proc. Am. Philos. Soc. vol. 44, 1905, p. 151.

[35] Mineralogical Abstracts. Mineralogical Society of London, Minerlog. Mag. and Jour., vol. 2, Nos. 1-12, 1923-1925, 1925, p.27.

[36] McKinstry, H. E., The Minerals of Rockport, Mass.: Am. Min., vol. 6, 1921, pp. 56-60.

Columbates of the rare earths were reported in the Cumberland region of Rhode Island. [37] Columbite was reported also in the King's River district, Calif.[38] An interesting occurrence of a tantalum mineral was discovered at a feldspar quarry at Henryton, Carroll County, Md., about 28 miles from Baltimore.[39] The tantalum was in irregular masses and rough crystals in the feldspar. It was not made use of, however, when the feldspar was being mined. An analysis of the ore gave the following results: tantalum oxide, 38.19 per cent; columbium oxide, 13.21 per cent; manganese oxide, 10.48 per cent; ferric oxide, 21.42 per cent; and silica, 12.98 per cent. Among the Idaho concentrates (mostly from gold placers) that he has examined, Shannon includes columbite and samarskite, as well as polycrase.[40] In the article referred to in the footnote, he has set forth methods and a determination table.[41]

FOREIGN SOURCES OF SUPPLY

Australia is by far the largest producer of tantalum minerals in the world, and Western Australia was the only division that recorded production before 1927, in which year the Northern Territory (now North Australia and Central Australia) reported a production of 168 pounds of tantalite, valued at £8. With the possible exception of Southern Rhodesia and Southwest Africa (of the Union of South Africa), there are no other known foreign sources of tantalum that are of other than scientific interest.

Western Australia

The Pilbara field is the principal producing center of Western Australia. Production in 1926 [42] was 19.45 tons, valued at £2,357; in 1927 the output fell to 15.28 tons, but with a higher value, £3,808; in 1928 [43] exports fell to 11 tons, valued at £2,749, or £250 per ton. Young and Moulden (a firm of Adelaide, in which Lady Hackett Moulden, formerly of Western Australia, is chiefly interested) within the last few years opened up the old pegmatite through the whole length of the claims M. L. 86 and 87 in the long-deserted Wodgina property. "High-grade tantalite is recovered, some from the soil on the slope below the outcrop and some from the lode. Concentrates are made at the rate of 20 to 30 tons per annum and are sold at about £200 per ton. They are almost clean manganontantalite, with a little spessartite and a few pebbles of scheelite."44/ Simpson, from whom the preceding quotation is taken, described the deposits of Western Australia as he saw them in 1927. A digest of his description follows.45/

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[37] Gedney, E. K., and Brown, C. W., Some New Occurrences of Rare Metals and other Minerals from the Cumberland Region of Rhode Island. Paper presented before the Eighth Annual Meeting of the Mineralogical Society of America, Dec., 1927.

[38] California State Mineralogist, Mining in California--Tantalum: Rept. of State Mineralogist, vol. 25, No. 3, July, 1929, p. 336.

[39] Pratt, Joseph Hyde, Work Cited, p. 1316

[40] Shannon, Earl V., Mineralogy of Some Black Sands from Idaho, with a description of the Methods Used for their Study: Proc.U.S. National Museum, vol.60, [illegible], 1921, 33 pp.

[41] Mineralogical Society of London, Mineralogical Abstracts: Mineralog. Mag. and Jour., vol. 2, Nos. 1-12, 1923-25, p. 362.

[42] Engineering and Mining Journal, Keen Interest Shown in Australian Tantalite: Vol. 126, No. 9, Sept. 1, 1928, p. 349.

[43] Holman, ________, Tantalum: Min. Jour., Jan. 25, 1930, p. 10

The Pilbara gold field is in the northwest part of Western Australia. It has coastal steamer service and the Perth-Derby air mail service. A railway 114 miles long runs from Port Hedland to Marble Bar in the middle of the gold field. Properties at Mud Springs, Tabba, and Wodgina are actively mined. At Mud Springs (Moolyella, a few miles northeast of Marble Bar) Ellis and Maher are mining "alluvium," which is crushed and concentrated. It consists of 3 feet of "wash", made up of "ferruginous clay and gravelly quartz and feldspar (microcline)." Above is 3 to 6 feet of "overburden," which is removed by tractor and scoop.

A tin concentrate (purposely incompletely dressed) contained 90.1 per cent cassiterite, with 1.5 per cent manganocolumbite, 8.3 per cent spessartite (manganese garnet), and traces of zircon, magnetite, monazite and epidote. With care it could be dressed up to 70 per cent metallic tin. About two-thirds of the concentrate was finer than 10 mesh.

In Moolyella gully there is a small patch of alluvium, at the bottom of which is 2 to 3 feet microcline quartz gravel "cemented with sand and tough green clay." Most of the first concentrates carry spessartite (specific gravity 4.2), some monazite (specific gravity 5.3), and manganocolumbite (specific gravity 5.7), besides cassiterite. The country rock is granite cut by thousands of pegmatites, only a few of which appear to carry visible cassiterite. Water is scarce.

The Tabba Tabba tin and tantalum field is about 8 miles southwest of the 35-mile post on the Marble Bar Railway. It appears to be very small and has been proved over an area of less than 4 square miles. The country rock is epidiorite, with granite close by on the west and south. No tantalite was observed in the principal workings, but at Tabba, Thelemann's M. L. 317, are at least two parallel pegmatites which have shed abundant gravelly tantalite on the gentle slopes. Only this detrital ore is now being worked. Dry concentration by hand sieving is used. "The soil is nowhere more than a foot or two thick, but tantalite can be obtained from almost every square yard of the lease." On Young and Moulden's M. L. 312 there is a prominent pegmatite "vein," up to 40 feet wide in places, bearing northwest. Soft detritus alongside this vein is being partly concentrated for tantalite by sieving, and the concentrates, running about 10 per cent tantalite, are being carted to Wodgina (58 miles) for final dressing.

There are three good wells close to the leases at Tabba, and abundant water is available from them at 40 feet for machine crushing and concentration but not for any large-scale sluicing. The old and new tantalite prospects on Strelling Station, north of the railway between the 30 and 35 mile posts, were not visited...

44/ Simpson, Edward S., Pilbara Tin and Tantalum Deposits: West Australian Chem. Eng. and Min. Rev., vol. 20, Melbourne, 1927, pp. 83-87.

45/ Hess, Frank L., Rare Metals--Tantalum: Mineral Resources of the United States, 1927, Pt. I. Bureau of Mines, 1929, pp. 408-409.

The main vein(at Wodgina) runs north-south and a little beyond the leases in both directions. It is mainly coarsely crystallized albite, with irregularly distributed bunches of quartz, microcline, and lepidolite and many small crystals of spessartite. Associated with the tantalite at the northern end are small quantities of mackintoshite, thorogummite, pilbarite, and hydrothorite, large masses of lithiophilite (8 to 9 per cent Li_2O), masses in the workings and in the dumps ranging from 1 to 300 pounds, and small veins and larger masses of apatite. A dump shows large masses of white beryl. In a coarse vein at the north end is a large mass lepidolite, and smaller masses are scattered along the main vein. Near the middle there is 4 or 5 feet of coarse albite on each side of a barren granite horse 20 feet thick. At this place tantalite in lumps weighing 1 to 5 pounds stud the albite thickly. Float tantalite shows on the surface of the leases from one end to the other, although 70 tons was obtained from the surface many years ago. The mineral from the vein is remarkably uniform in grade and averages Ta_2O_5 68 per cent, Sb_2O_5 14 per cent, MnO 13 per cent, and FeO 2 per cent. Buyers want none under 63 per cent Ta_2O_5. The Mount Cassiterite mine, M. L. 84, has SnO_2 in the chlorite-biotite schist alongside the pegmatite. On one dump spodumene is rather common. There is very little alluvial ground in the Wodgina area.

Mount Francisco is 16 miles south-southwest of Wodina, but 33 miles by road. Four miles east of Francisco well is Hooley's columbite lode. It is an east-west pegmatite, principally albite with quartz, microcline, and in places muscovite and lepidolite. Baryl is common in crystals ranging from a few ounces to 50 pounds. Mangano-columbite is abundant in the vein and in the surrounding soil. Several tons of pebbly ore (¼ inch to 3 inches) could be easily collected on the surface, but the clean concentrate carries an average of only 22 per cent Ta_2O_5 and 58.2 per cent Cb_2O_5. The minimum is 13.7 per cent Ta_2O_5 with 65.9 per cent Ob_2O_5 and the maximum, 44.7 per cent Ta_2O_5 with 37.1 per cent Cb_2O_5. No SnO_2 was seen. The country rock is granite. Five miles south of Francisco well G. J. Hooley has discovered float cassiterite in considerable quantity. Good prospects (2 to 3 ounces from 10 pounds) have been obtained in the soil along the outcrops of two east-west pegmatites. The concentrates carry a little monazite and euxenite. A basic dike not far away contains ceylonite.

In some of the workings at Cooglegong cassiterite with "gravelly monazite" is found. An analysis showed 4.5 per cent ThO_2 and 55 to 65 per cent Ce_2O_3. Three tons of gadolinite were found in a pegmatite 4 miles east of the Cooglegong Hotel and a half mile west of Black Ridge. None is now visible in the pegmatite, but gravel from a dry stream bed gave cassiterite 30 per cent,. gadolinite 30 per cent, magnetite 25 per cent, fergusonite 10 per cent, and manazite, garnet, and limonite 5 per cent.

The following table shows production in Western Australia.

Quantity and value of tantalite in Western Australia during 1928 and totals to date, as reported to the Mines Department[1]

Locality	Number of lease, claim or area	Registered name of company or lease	1928		To date	
			Tons	Value	Tons	Value
Pilbara Goldfield (Marble Bar Dist.):						
Tabba Tabba	M.L. 317	Koolinda North	1.00	£250	1.00	£250
Do.	M.L. 321,322	Strelley North leases	.57	165	.57	165
Wodgina	M.L. 86,87,95	H. M. and Anchorite leases	7.19	1,793	93.22	15,813
Do.	M.L. 293	May Be	-	-	2.00	240
Do.	-	Sundry claims	-	-	51.30	6,124
			8.76	2,213	148.29	22,592
Greenbushes Mineral Field:						
Greenbushes	(369)	Enterprise			3.19	£1,804

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[1] Report of the Department of Mines, Western Australia, for the year 1928, Mining Statistics Section, Perth, 1929, p. 55.

North Australia

A government publication [46] reports that there are now good demand and good prices for tantalite ore containing over 45 per cent tantalic acid, and that increased interest is being taken in mining for tantalite. Production for the year ending June 30, 1929, was 9 cwt. 2 qrs., valued at £11,436 5s. 8d. For the previous year it was 1½ cwt., valued at L8.

What is claimed to be the biggest deposit of tantalite in the world exists on the property of Messrs. Gitsham and Johnson (65 miles from Port Darwin); it is reported to have been taken over, under option for four months, by the Mutooroo Copper Co.[47] The Industrial Australian and Mining Standard of January 30 gives the report of its mining expert, as follows: "The deposit includes all Mount Finniss and the surrounding slopes. Exhaustive propsecting has proved an alluvial area of 60 acres, with an average depth of 2 feet. The average yield of ore is 2 ounces to the dish, equal to £10 per yard. The tantalite available in the area is estimated at approximately 790 tons. The lode is 100 yards wide, and it shows free tantalite.

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[46] Report on the Administration of North Australia for the Year Ended June 30, 1928, Canberra, 1929, p. 11.

[47] Industrial Australian and Mining Standard, Tantalite in North Australia, vol. 83, No. 2130, Feb. 13, 1930, p. 115.

It has not been developed, only prospecting work having been done."[48]

A report from Darwin in April, 1929, is to the effect that an option over a tantalite claim at West Arm, owned by P. Lugg and V. Litchfield, was secured by Young and Moulden, in the name of Lady Moulden. Miners in West Arm, it is reported, secured from a dike formation 5 pounds of tantalite, which assaygd 73 per cent.

Other Sources

Bavaria (Europe).--Müllbauer [49] has continued the work of Laubmann and Steinmetz on phosphate-bearing pegmatites of Hagendorf, in Bavaria. "Primary minerals of the liquid-magmatic phase include columbite," with others. "A subsequent hydrothermal phase gave rise" to columbite (thin platy crystals), among other minerals.[50]

Belgian Congo (Africa).--Buttgenbach [51] gives columbite as one of the concentrates from the Lukulu River.[52]

Brazil (South America).--Columbite and samarskite, together with monazite, occur about a league south of Divino de Uba (field of Divino, town of Uba), Minas Geraes, Brazil. These three minerals are associated in the deposit in radial aggregates. The center is generally samarskite, with columbite and monazite in crystallographic continuity.[53] The following analyses are given:

Columbite

Ta_2O_5	22.80
Cb_2O_5	59.04
FeO	15.08
MnO	3.36
CaO	trace
MgO	trace

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[48] Industrial Australian and Mining Standard, Tantalite in the Northern Territory: Vol. 83, Jan. 30, 1930, p. 75.

[49] Müllbauer, F., Die Phosphatpegmatite von Hagendorf i. Bayern (Neue Beobachtungen): Ztschr. Krist., vol 61, 1925, pp. 318-336.

[50] Mineralogical Society of London, Mineralogical Abstracts: Mineralog. Mag. and Jour., vol. 2, Nos. 1-12, 1923-1925, p. 417.

[51] Buttgenbach, H., Description des minéraux du Congo belge (Sixième mémoire): Mém. Acad. R. Belgique, Cl. d. Sci., 1923, ser. 2, vol. 7, 36 pp.

[52] Mineralogical Society of London, Mineralogical Abstracts: Mineralog. Mag. and Jour., vol. 2, Nos. 1-12, 1923-25, p. 265.

[53] Guimarãez, Djalma, Breve Noticia sobre una Jazida de Samarskita, Columbita, e Monazita: Servico Geol. e Mineral. do Brazil, Bol. 13, 1925, pp. 115-118.

Samarskite

	Specimen 1	Specimen 2
Ta_2O_5	20.18	21.26
Cb_2O_5	32.16	33.12
TiO_2	2.00	1.50
$(Y,Er)_2O_3$	12.24	15.90
ThO_2	1.16	0.66
UO_2	18.14	14.32
CaO	1.28	1.19
MgO	0.74	1.03
ZrO_2	2,15	1.12
FeO	7.95	7.58
H_2O	1.78	1.56

Canada (North America).-Although columbite-tantalite groups are well represented in Canada, tantalum has not been recognized in quantities of commercial importance, and there is no production. A few occurrences of mineralogical interest have been described, however. Columbite in Lyndoch Township, Renfrew County, Ontario, was described by Miller.[54] Columbite occurs, with hatchettolite and cyrtolite in a feldspar quarry at Hybla, on lot 17, Concession 8, Monteagle Township, Hastings County, Ontario.[55] The columbite is massive and very fine-grained. Ellsworth says that the feldspar from mines near Hybla containsthe most promising uranium minerals, not only for their uranium, but for columbium and tantalum, as well as other constituents, if they are recovered and marketed as pure compounds.[56] At New Ross, Lunenburg County, Nova Scotia, the mineral is found in the form of crystals often weighing several grams, in a coarse pegmatite that cuts an even-grained granite. Although the crystals are generally well formed, they are not bright enough for crystallographic measurements.

Walker and Parsons,[57] in the analyses given of the columbites indicated above, places the percentages of columbium oxide and tantalum oxide, respectively, as follows: Renfrew County, 55.79 and 15.21; Hastings County, 66.60 and 1.74; and New Ross, 63.08 and 14.48.

The Canadian Mining Journal recently contained the following statement:[58]

> R. Sweezey reports the discovery of tantalite showings to the northeast of Star Lake, in the Bird River district of southeastern Manitoba. The tantalite is said to occur in white, almost mica-free, feldspar bodies in the sediments that resemble veins more than dikes. Tourmaline and beryl are present. Steps to investigate their importance are under way.

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[54] Miller, Willet G., Economic Geology of Eastern Ontario; A Mineral of the Columbite Group: Bureau of Mines (Ontario) Rept. No. 7, 1897-98, pp. 234-237.

[55] Walker, T.L., and Parsons, A.L., Notes on Canadian Minerals--Allanite, Axinite, Columbite, and Sillimanite: Univ. of Toronto Studies, Geol. Series No. 16, Toronto, Ontario, Canada, 1923, pp. 35-36.

[56] Ellsworth, H.V., The Rare-Element Minerals of Canada: Canadian Chem. and Met., vol. 8, No. 11, Nov., 1924, p. 262.

[57] Walker, T.L., and Parsons, A. L., Work cited.

[58] Canadian Mining Journal, Tantalite Reported: Vol. 51, Mar. 28, 1930, p. 306.

Ceylon (Asia) — Professor Dunstan gives the following analysis of a fragment of columbite from Ceylon, the exact origin of which is unknown.59/

	Per cent
Ta_2O_5	7.30
Cb_2O_5	67.35
TiO_2	2.90
CaO	0.36
Fe_2O_3	2.05
MnO	10.30
FeO	9.22

He reports also a mineral allied to samarskite, two specimens of which—one weighing 1.2 grams (specific gravity, 5.50) and the other weighing 1.5 grams (specific gravity, 4.95)—were identical with specimens previously received and called annerodite, but which were shown to be fergusonite. Fergusonite, according to the reports on the results of mineral surveys,60/ is a very noticeable constituent of many of the gravels. The specimens analyzed at the Imperial Institute contained about 40 per cent of oxides of yttrium and the allied metals and from 2 to 2½ per cent of thoria.

Federated Malay States (Asia).—Strüverite was discovered in 1909 by R. L. Corbett in concentrates from the Sungei Sebantum, Salak North, Kuala Kangsar district, Perak. It came from a shallow alluvial tin mine, where the strüverite was a hindrance. Evidence seemed to point to a kaolinized granitic intrustion in hardened black shales as the original source of the mineral. The following analysis was made by S. J. Johnstone:61/

	Per cent
TiO_2	45.74
Ta_2O_5	35.96
Cb_2O_5	6.90
FeO	8.27
MnO	trace
SnO_2	2.67
SiO_2	0.20
H_2O at 105° C.	0.08
H_2O above 105° C.	0.42
	100.24

Although, according to Scrivenor,62/ inquiries have been made concerning the feasibility of working the strüverite for its tantalum content, doubt of its proving profitable has been increased by the planting of the land with rubber, for which the landowner would have to be compensated before the work could be undertaken.

59/ Dunstan, W. R., Report of the Results of the Mineral Survey in 1904-05: Ceylon Mineral Survey; Colonial Reports-Miscellaneous No.37, London 1906, pp.30-31.

60/ Dunstan, W. R., Work cited, p. 40.

61/ The Mineral is described in detail by Crook, T., and Johnstone, S. J., on Strüverite from the F.M.S.: Min.Mag., 1912, pp. 224-231.

62/ Scrivenor, J. B., The Geology of Malayan Ore-Deposits, London, 1928, p. 146.

Finland (Europe).–References are made in footnotes to descriptions of tantalum-bearing minerals in Finland.[63]

France (Europe).–A number of different tantalum-bearing minerals have been identified as minor constituents of pegmatites in various localities of France: neotantalite (57.70 per cent tantalum oxide and 22.00 per cent columbium oxide) in Allier, a Department of central France;[64] skögbolite (82.90 per cent tantalum oxide and traces of columbium oxide) in Haute-Vienne, a Department of west-central France;[65] and yttrotantalite in Tarn-et Garonne, a Department of southern France.[66] Lacroix discusses at some length the columbites and tantalites in the Department of Haute-Vienne, which occur in the pegmatites of the region of Chanteloube;[67] he discusses also manganocolumbite, manganotantalite, and microlite.[68]

French West Africa.–Pyrochlore is reported[69] in the Los Islands, on the northwestern coast of Africa, in microscopic crystals in nepheline syenite; in great abundance as microscopic particles in barkevikite on the Island of Kassa; and in aegerine on the Island of Rouma.

Greenland (North America).–A French writer,[70] in a review of tantalum, says that the Pennsylvania Salt Co. found considerable tantalum in the cryolite imported from Greenland. A columbite from Greenland, the analysis of which is quoted by Dana,[71] has a specific gravity of 5.395.

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[63] Gahn, J. Gottlieb, Berzelius, Jac., and Eggertz, H. P., Tantalmetallens egenskaper, halten af syre i dess oxid, dennes mattningscapacitet och kemiska egenskaper: Afhandlingar i Fysik, Kemi och Mineralogi, vol. 4, 1815, pp.262-293.

Gahn, J. Gottlieb, Berzelius, Jac., and Eggertz, H. P., Eigenschaften des Tantal-Metalls, Sauerstoffgehalt seines Oxyds, seine Sattigungscapacitat und chemische Eigenschaften: Schweigger, S.S.C. Jour. Chem. Ph., vol. 16, 1816, pp. 447-475.

Gahn, J. Gottlieb, Berzelius, Jac., and Eggertz, H. P., Des recherches de M. Berzelius, propriétés du tantale, etc.: Ann. Chim. Phys., vol. 3, 1816, pp. 146-160.

[64] Lacroix, A., Mineralogie de la France et de ses colonies: Vol. 4, Paris, 1910, p. 606.

[65] Lacroix, A., Work cited, p. 618.

[66] Lacroix, A., Work cited, p. 612.

[67] Lacroix, A., Work cited, pp. 616-617.

[68] Lacroix, A., Work cited, pp. 613-615.

[69] Lacroix, A., Work cited, pp. 603.

[70] Breuil, Pierre, Propriétés, métallurgie et emploi du tantale: La Génie Civil, vol. 54, 1908, pp. 7-8 and 25-27.

[71] Dana, Edward S., A System of Mineralogy, John Wiley & Sons, New York City, 1892, p. 733.

India (Asia).—Columbite has been known to exist in India, especially in the Gaya and Hazaribagh districts and in Mysore.72/ Those of Gaya are the most radioactive. A number of analyses had been made for the determination of radium in ores in the Gaya district, but no complete analysis had been attempted. N. C. Nag, assistant director of Bose Institute, Calcutta, therefore made an investigation of samples of columbite from the district; M. K. Ray supplied specimens weighing from 30 to 60 grams. The following is the result of the chemical analysis, no result being recorded here of the other tests:

	Per cent
Columbium pentoxide	59.05
Tantalum pentoxide	19.85
Ferrous oxide	7.90
Ferric oxide	0.22
Manganous oxide	11.62
Cupric oxide	0.15
Uranium oxide (U_3O_8)	0.80
Moisture	0.24
Oxides of silicon, lead, and bismuth	Traces only

This is representative of a large number of concordant estimations. Both the chemical and the electroscopic methods of estimations gave the same amount of uranium present in the columbite; and the other constituents present conformed fairly to its being 25 per cent tantalate and 75 per cent columbate (niobate). Its somewhat low specific gravity was not exceptional and was probably owing to its high manganese content.

Crystals and lumps of columbite are found in the pegmatite of Mysore, particularly in the mica pits. The mineral is nearly black, has a submetallic luster, and is composed of niobate and tantalate of iron and manganese. Licensees produced several hundred tons from pits near Yelwal and Tagadur, in the Mysore district, and reported it as tantalite. A sample from Tagadur was found to contain only 1.14 per cent of tantalic acid—the small percentage proving it to be columbit

In the decomposed pegmatite near Bangalore small pieces and plates of what appeared to be samarskite were found associated with monazite. 73/

A more detailed description of the occurrence of the columbo-tantalates of India is given by Das-Gupta.74/

72/ Nag, N. C., Chemical and Electroscopic Examination of a Sample of Radioactive Columbite from Gaya District: Trans. Geolog. Min., and Met. Soc. of India, Calcutta, vol. 2, No.1, Mar., 1929, pp. 1-6.

73/ Smeeth, W. F., and Iyengar, P. Sampat, Mineral Resources of Mysore: Gen. Series, Bull. No. 7, Dept. of Mines and Geol., Mysore State, Bangalore, 1916, p. 192.

74/ Das-Gupta, Hem Chandra, Indian Mineral Industry: Rept. of the Indian Assoc., for the Cultivation of Science, and Proc. of the Science Convention for the year 1918, Calcutta, 1920, pp. 166-169.

Japan (Asia).—Mineralogical abstracts [75] makes reference to samarskite from Ishikawa, Province of Iwaki, an analysis of which showed 41.11 per cent niobium pentoxide and 10.00 per cent tantalum pentoxide. A new mineral, ishikawaite, from the same district, is described as resembling samarskite. Crystals of columbite, from the same locality, showed 78.94 per cent $(Nb,Ta)_2O_5$. Fergusonite from Naegi, Province of Mino, gave 43.77 $(Nb,Ta)_2O_5$. "In Naegi, fergusonito is found with cassiterite, topaz, wolframite, naegite, and other minerals in the river sand in the biotite-granite region and is about 3 millimeters in diameter of the cross section and 5 to 8 millimeters in length." [76] Naegi was the only known locality producing fergusonite until the summer of 1924, when Mr. Watase (then a geologist of the Imperial Geological Survey) collected a crystal of the mineral at Hakatamura, Ochigori, Province of Iyo. Hakatamura is approximately 7 kilometers northwest of the town of Imaharu, in the northern part of the Takanawa Peninsula, in Shikoku. The mineral occurs in pegmatite dikes through biotite granite. The following excerpt is taken from the description given by Sato: [77]

> The dikes are generally lenticular in shape and measure 2 to 5 meters in width and about 20 meters in length. The middle part of the dike consists of large anhedral crystals of quartz and orthoclase, with a subordinate amount of biotite and pyrite, while the marginal part is composed of granular aggregates of quartz and feldspar, with a small amount of biotite. The texture is often extremely coarse, and there is a frequent tendency to the graphic structure. The dikes run in the direction of east-west and are steeply inclined. The crystals are imbedded either in orthoclase, biotite, or quartz, or are found in their interspaces, together with allanite, pyrite, chalcopyrite, and some variety of zircon (?). Those which are imbedded in orthoclase or found in the interspaces of orthoclase and biotite or quartz are usually large in size, attaining 2 or 3 centimeters in length and 5 or 6 millimeters in width, and well crystallized, while those that are imbedded in biotite are small in size and often lenticular in shape. Crystals are usually very acute pyramidal or long prismatic in habit. . . .

Mr. Kimura, [78] assistant professor of the Tokyo Imperial University, made the following analyses of the Naegi and the Hakatamura fergusonite:

Hakatamura		Naegi	
MgO	0.58	MgO	trace
CaO	1.40	CaO	1.16
Al_2O_3	1.35	Al_2O_3	0.20
Fe_2O_3	0.66	Fe_2O_3	3.97
Nd_2O_3, etc.	0.89	Nd_2O_3, etc.	1.99
Y_2O_3, etc.	40.39	Y_2O_3, etc.	37.64

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[75] Mineralogical Society of London, New Minerals: Mineralogical Abstracts, Mineral. Mag. and Jour., vol. 2, Nos. 1-12, 1923-1925, pp. 9 and 36.

[76] Sato, Denzo, Brief Notes on Fregusonite and Allanite from Iyo, Shikoku; Fergusonite: Koto Commemoration Volume, Tokyo, 1925, p. 49.

[77] Sato, Denzo, work cited, pp. 49-50.

[78] Sato, Denzo, Work cited, pp. 50-51.

Hakatamura (Contd)

SiO_2	0.79
TiO_2	1.15
SnO_2	0.35
UO_2	3.18
$(Cb,Ta)_2O_5$	44.97
CO_2	0.35
H_2O	3.92
Total	99.98

Naegi (Contd)

SiO_2	2.83
TiO_2	1.65
UO_3	0.85
$(Cb,Ta)_2O_5$	43.77
ThO_2	2.91
CeO	1.26
Loss below 110°	0.24
Loss above 110°	1.85
	100.32

In the proceedings of the Third Pan-Pacific Scientific Congress, Sato[79] mentioned the fergusonite in the river sand in the biotite-granite region of Naegi (Province of Mino) and in the pegmatite of Hagatamura (Province of Iyo), samarskite and ishikawaite in the pegmatite of Ishikawa-yama, and columbite in the pegmatite of Ishikawa-yama and Yamano-o, at the northern foot of Mount Tsukuba (Province of Hitachi).

Madagascar.- Various tantalum minerals, including columbite, more or less tantaliferous, have been reported in different parts of Madagascar, where they are found especially in association with radium ores at Ampangabe.

According to Lacroix,[80] ampangabeite is found with beryl, columbite, strüverite, and monazite in pegmatite at Ampangabe. It occurs in rectangular prisms "some centimeters long" in groups with roughly parallel axes. An analysis made by M. Pisani gives columbium oxide 34.80 per cent and tantalum oxide 8.90 per cent. Fergusonite[81] is found in pegmatite between Tamatave and Beforona. An analysis of M. Pisani places the columbium oxide at 50.10 and the tantalum oxide at zero. Lacroix reports betafite, also, as follows:[82]

> Under the name of blomstrandite, a mineral was described, which occurs with rose feldspar in pegmatite at Ambolotara, near Betafo. Later the name of betafite was given to this mineral, and another mineral was called blomstrandite, which is found in octahedra, 4 centimeters in greatest dimension, in red earth with beryl, black tourmaline, almandite, magnetite, and cyrtolite at Tongafeno. It is found also in pegmatites at Antandrokomby, Tsilaisina, Maharitra, Antsongombato, and in large crystals at Manendrika, near Antanamalaza.

M. Pisani's analysis gives columbium oxide as 23.30 per cent and tantalum oxide as 28.50 per cent. The same author [83] reports samarskite in small quantity in pegmatite with beryl, almandite, magnetite, and other minerals at Manendrika, a specimen of which when analyzed by M. Pisani gave 43.60 per cent of columbium oxide and 11.15 per cent of tantalum oxide. Lacroix has also identified samiresite at Samiresy, southwest of Lake Tritriva, where it occurs in a red earth resulting from the decomposition of a pegmatite, and where it is associated with brown zircon, euxenite, bismuth,bismuthite, and pyromorphite. Pisani's analysis gives 45.80 per cent columbium oxide and 3.70 per cent tantalum oxide.[84]

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[79] Sato, Denzo, Some Minerals Containg the Rarer Elements: Proc. 3d Pan-Pacific Sci. Congr., Tokyo, 1926, vol.1,1928, pp. 865-866. Chem. Abs., vol. 23, No. 19, Oct. 10, 1929, p. 4650.

[80] Lacroix, A., Mineralogie de la France et de ses colonies, vol.5, Paris, 1910,p.3

[81] Lacroix, A., Work cited, vol. 4, pp. 608-609.

[82] Lacroix, A., Work cited, vol. 4, pp. 612-613; vol. 5, pp. 72, 73, and 92.

[83] Lacroix, A., Work cited, vol. 5, pp. 69-70.

Ungemach [85] gives crystallographic descriptions of minerals of pegmatite origin, among which are strüverite, euxenite, and columbite. [86]

Namaqualand (Africa).-Tantalum and columbium were first discovered in Namaqualand (German Southwest Africa) in August, 1911, when specimens of columbite and tantalite from the Richtersveld and other parts of Namaqualand were identified. Since 1911 numerous occurrences have been discovered in the pegmatites of the granite area, and a large amount of prospecting has been done. The minerals are associated with lithium, bismuth, and manganese. "The proportion of tantalum to columbium varies very considerably and is roughly indicated by the specific gravity of the mineral, the highest gravity coinciding with the largest proportion of tantalum. The heaviest specimen examined had a gravity of 7.5 and was found to be manganotantalite, containing 82.4 per cent of combined oxides of tantalum and columbium. It is likely that the amount of tantalum oxide in this specimen would be over 80 per cent. Another specimen (part of a very large crystal) had a specific gravity of 6.85 and contained 82.2 per cent of the combined oxides. When the gravity is as low as 5.4, very little tantalum is present. . . It is likely that important alluvial deposits will be discovered in Namaqualand, since the mineral is very heavy and does not weather easily."[87]

The South African Mining and Engineering Journal [88] in 1929 said that there were possibilities of the production of tantalite from the pegmatites of Namaqualand, which have been shown to contain massive beryl, suggesting that if in the course of beryl mining any appreciable quantity of tantalite can be recovered it will find a market at good prices.

Norway (Europe).-Columbite was first discovered in Norway in 1879, when a good crystal from the pegmatites at Annerod, in Smalenene, was sent to the mineral collection of the university and subsequently identified as columbite by Jameson,[89] who says that later investigations proved that in the pegmatites in Smalenene so also in Valer (east of Moss) and in Rade and Rygge columbite is one of the most easily accessible of minerals; that in other districts it appears to be relatively sparsely distributed with the granite pegmatites. The locations of the best known of the Norwegian deposits are as follows:[90]

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[84] Lacroix, A., Work cited, pp. 71-72.

[85] Ungemach, H., Contribution a la minérlogie de Madagascar: Bull. soc. française min., vol. 39, 1916, pp. 5-38.

[86] Mineralogical Society of London, Mineral Abstracts: Supplement, Mineral. Mag. and Jour., vol. 2, Nos. 1-12, 1923-25, p. 269.

[87] Versfeld, Wm., The Base Metal Resources of the Union of South Africa: S. A. Dept. of Mines and Ind. Mem. No. 1, 1919, 107 pp.

[88] South African Mining and Engineering Journal, Money and Mines:- A Tantalite Possibility: vol. 40, Pt. I, No. 1969, June 22, 1929, p. 3.

[89] Brogger, W. C., Bemerkungen über die einzelnen Mineralspecies der sudnorwegischen granitischen Pegmatitgange; I. Niobate, Tantalate, Titanate und Titanoniobate (Jameson--Columbit--Niobit): Videnskabe-Selskabet Skrifter, Math.-Naturv. Klasse 1906, No. 6, Kristiania, 1906, p. 53.

1. Annerod, in Valer, east of Moss
2. Odegardssletten, in Valer
3. Kjaersund, in Valer
4. Dramstad, Island of Faeo, in Vandsjo
5. Island of Oxeno, in Rade
6. Huggenaeskilen, in Rygge, south of Vandsjo
7. Grevsrud, in parish of Rygge
8. Hullingsas, in parish of Rygge
9. Sameja, in parish of Rygge
10. Aveno, between the fiords of Krakstad and Kure
11. Fuglevik, near the fiord of Krakstad
12. Lorebo, near Aker
13. Berg (?), in Rade
14. Elvestad
15. Karlshus
16. Halvorsrod
17. Starengen
18. Lundeby
19. Skato, Sanokedal, Bamle (in the neighborhood of Kragero)
20. Ramskjaer, Sondeled, near Risor
21. Baseland
22. Rostol, near Arendal
23. Kristiansand (neighborhood of)
24. Havas Bruch, Eftevand
25. Hittero

Brøgger gives crystallographic descriptions and analyses of columbite;[91] in addition he discusses fully the fergusonite,[92] samarskite,[93] euxenite, and polychrase [94] of the same regions in Norway. Supplementary notes on fergusonite, ilmenorutile, columbite interwoven with samarskite, euxenite, and priorite (new analysis), a continuation of the work of Brøgger in 1906, are published by Jakob Schetelig.[95] The new fields are mostly from the feldspar quarries in the parish of Iveland, Saetersdalen.

Rhodesia (Africa).-The South African Mining and Engineering Journal[96] reported as follows:

> There is quite a small "boom" on in tantalite as the result of American inquiries, and a deal of pegging has taken place in the vicinity of the well-known ebonite and tantalum claims. The properties owned by the Rhodesian Tantalum Syndicate and Mr. A. J. A. Miller are under option, and development work is proceeding. An unusual and very interesting tantalite discovery has been made on the Conwar blocks. The tantalite has been found

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[90] Brøgger, W. C.,Work cited, pp. 53-59.
[91] Brøgger, W. C.,Work cited, pp. 59-81.
[92] Brøgger, W. C.,Work cited, pp. 31-38.
[93] Brøgger, W. C.,Work cited, pp. 138-152.
[94] Brøgger, W. C.,Work cited, pp. 82-97.
[95] Schetelig, Jakob, Ergänzungen zu Band I, Niobate, Tantalate, Titanate and Titano-niobate, pp. 139-148.
[96] South African Mining and Engineering Journal, Tin in the Fort Victoria District vol. 40, Pt. I, May 18, 1929, p. 332.

in reef; the reef is from 8 to 15 feet wide, and in sections carries up to 10 per cent of tantalite. An analysis of the concentrate recovered by a small trial crushing gives: tantalum pentoxide, 69.25 per cent; columbium pentoxide, 3.50 per cent; silica, 4.40 per cent; stannic oxide, 33 per cent; ferrous oxide, 4.01 per cent; alumina, 1.03 per cent, manganous oxide, 12.95 per cent; lime, 2.54 per cent; magnesia, 1.82 per cent. Samples of these concentrates submitted to American buyers resulted in an offer of $1 per pound containing Ta_2O_5.

In 1911 F. Sinclair-Ross recognized tantlite in the Victoria tin field, which lies halfway between Mara Hill and Lemoenfontein, in the Ndanga district. (For the geology of the Victoria tin field, see footnote reference below.[97]) The tantalite lies in the sheet of 'rubble' formed by the disintegration of a highly quartzose permatite, ungreisenized, but in the immediate vicinity of bodies of cassiterite-bearing lepidolite greisen." For further description of the occurrence, see article by Zealley,[98] from which the preceding quotation and the following excerpt are taken:

> The density values of the Victoria tantalite considered in conjunction with the results of blowpipe tests suggest that the mineral is of varying composition somewhere near the tantalite end of the columbite-tantalite series, that is, an appreciably stanniferous tantalite containing about 65 to 70 per cent of tantalum oxide (Ta_2O_5) and not much more than 10 to 12 per cent of columbium oxide (Cb_2O_5). The highest value (7.301) obtained in two instances is that of manganotantalite.
>
> The associated minerals detected include quartz exteriorly intergrown, fine pearly-lustered whitish mica, also slightly intergrown, and inconspicuous crystalline crusts and pockets of a brown pyrochlore, tending to be limited to what were free surfaces of the tantalite aggregates but partly intergrown. . . The tantalite is virtually free from inclusions. In one specimen parallel intergrowth and embedded grains of an unidentified mineral occurred at the core of a radiated lump....

Mennell [99] says that the tantalite from this locality has the peculiarity of containing a considerable amount of combined tin oxide, and that this has proved a stumbling block in the way of marketing the ore. A Government publication of Southern Rhodesia reports the production of 1 short ton of tantalum, valued at £71, in 1926, as the full amount produced to date.[100] Northern Rhodesian official publications report no production of tantalum.

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[97] Maufe, H. B., Report of the Director, Geological Survey of Southern Rhodesia, Salisbury, 1911. pp. 31-38.

[98] Zealley, A. E. V., Notes on Newly Recorded Rhodesian Minerals: Proc. Rhodesian Sci. Assoc. vol. 16, pt. 1, June, 1917- May, 1918, Bulawayo, 1917, pp. 20-23.

[99] Mennell, F. P., The Steel Hardening Group of Metals: Rhodesia Resources Committee, Bulawayo, 1921, p. 124.

[100] Rhodesia Chamber of Commerce, Report of Executive Committee on Output of Gold and other Metals and Minerals for the Month of December, 1929, Bulawayo, 1930. 7 pp.

Zealley [101] refers to tantalite (discovered in 1916) 4½ miles southwest of Odzi Siding, Makoni district, blowpipe tests of which indicate that the mineral is probably manganotantalite free from tungsten and tin. He describes, also, [102] microlite of the lithia greisen bodies at Odzi, in the neighborhood of which tinstone and another tantalum mineral occur, although they are not actually associated with it. A more detailed description of the tantalite of the Odzi region is given by Zealley in an article on the geology and mineral deposits of the Umtali Gold Belt.[103]

Russia (Europe).- Cernik [104] describes fergusonite of the Blum mine, Ilmen Mountains, of the Urals. Specimens showed the associated minerals to be samarskite, ilmenorutile, tourmaline, perhaps malacon, and two undetermined minerals intergrown with the samarskite. Analysis gave 47.12 per cent niobium pentoxide and 5.10 per cent tantlum pentoxide.[105]

P. Walther [106] described a bright greyish yellow crystalline powder, consisting of 98.5 per cent of tantalum, about 1.5 per cent of columbium, with 0.001 per cent of manganese. The specific gravity of the substance, which was obtained in a gold dust found in the Ural Mountains, is 9. Walther said that this substance may be considered native tantalum, and that there is no other record about the occurrence of native columbium or native tantalum.[107]

Russia (Asia).-A small quantity (a few dekagrams) of native tantalum from the Altai Mountains was taken from the collection of a deceased mining director. At about the same time a specimen of tantalum from an undefined gold-washing locality in the Urals was found under similar circumstances. W. von John [108] says:

> The Altai tantalum contains gold, from a slight trace to 0.0095 per cent, but no trace of manganese, tin, and niobium could be detected; the latter three have been found in the Ural tantalum. The average percentage of tantalum is 98-99 per cent. The average measurement of the crystals is about 0.1 mm., and the crystals are of the regular system, as in the Ural tantalum. The hardness (between 6 and 7) and the specific gravity (11.2) are the same.

Smyrna (Asia).- Columbite occurs in quartz veins near Darmanlar.[109]

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[101] Zealley, A. E. V., Work cited, pp. 22-23.

[102] Zealley, A. E. V., Work cited, pp. 20-21.

[103] Zealley, A. E. V., Note on the Geology and Mineral Deposits of the Umtali Gold Belt: Southern Rhodesia Geol. Survey, Short Rept. 2, 1918, pp. 3-4.

[104] Cernik, G. P., La consistence chimique d'une varieté de la fergusonite d'Oural: Bull. Acad. Sci. Russie, vol. 15, ser. 6, 1921, pp. 419-424.

[105] Mineralogical Society of London, Mineralogical Abstracts: Mineral. Mag. and Jour., vol. 2, Nos. 1-12, 1923-25, p. 405.

[106] Walther, P., Nature, Sept. 16, 1909, p. 335.

[107] Das-Gupta, Hem Chandra, Indian Mineral Industry: Rept. of the Indian Assoc. for the Cultivation of Science and Proc. of the Science Convention for the Year 1918, Calcutta, 1920, p. 165.

[108] Von John, W., Native Tantalum: Nature, vol. 83, London, June 2, 1910, p. 398.

[109] DeLaunay, L., La Gèologie et les riches minérales de l'Asie, Paris, 1911, p. 637.

Sweden (Europe).- Gahn and Berzelius 110/ describe a large number of minerals from Finbo, including yttrocerite, cassiterite carrying 2.4 per cent tantalum oxide, and tantalite carrying 16.75 per cent tin oxide. They describe also tantalum from Broddbo, carrying 6.12 per cent WO_3 and 8.40 per cent SnO_2.

Union of South Africa.- Little more than specimens have been discovered in the Union of South Africa, though, according to the South African Mining and Engineering Journal, 111/ an appreciable sum has been spent in looking for signs of an economic deposit in Southwest Africa (a mandate of the Union), specimens from which have intrigued mineral brokers, exporters, and others. However, Government reports 112/ indicate that the only production in Southwest Africa was that of 0.3 long ton in 1928. However, Holman 113/ says that orders for the mineral were accepted in 1927. Fresh discoveries of very high-grade ore were reported in 1928. Samples submitted were remarkable for their low content of columbium. It was stated that 5 tons monthly of material containing over 65 per cent of tantalum oxide (Ta_2O_5) could be guaranteed and that orders were being made by cable. A firm offer from London for a trial shipment at 2s. a pound, c.i.f. Europe, had not been accepted by December, 1929. A price of 1s. 9d. per pound, on a basis of 65 per cent, was made for shipment, via Walfish Bay, but no deliveries were made.

Wagner 114/ mentions columbite at Dawib (Southwest Africa) as being one of the constituents of the pegmatites, which are generally muscovite-bearing. Pisar 115/ reports tantalite in crystals and crystalline masses, up to three-fourths inch in diameter, in the Karib district, at Donkerhoek, 33 miles east-southeast of Jackalswater. Reunig 116/ describes pegmatites with tantalum, niobium, and rare earths, with tantalite, columbite, and blomstrandite.

IMPORTS AND EXPORTS

There were no imports of tantalum or tantalum ores recorded in 1918, 1919, 1922, or 1924. In 1920 there were imported 60 pounds of ferroalloys (tantalum), valued at $54; and in 1921, 1,229 pounds of ferroalloys (tantalum), valued at $788, and 6,785 pounds of steel-hardening ores (tantalum), valued at $1,327. Beginning with 1923, the classification is "Ores of ferroalloying metals (tantalum)" and the

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110/ Gahn, J. Gottlieb, and Berzelius, J., Untersuchungen einiger in der Nähe von Fahlun gefundenen Fossilien: Jour. Chem. Ph., Schweigger, Dr. J. S. C., vol. 16, 1816, p. 241.

111/ South African Mining and Engineering Journal, Tantalite in Southwest Africa: Vol. 38, Pt. II, Jan. 7, 1928, p. 502.

112/ Totten, Ralph J., (American consul general, Cape Town), Mineral Production of the Southwest African Protectorate in 1928: Consular Rept., May 29, 1929, Bureau of Mines Foreign File 5952.

113/ Holman, Tantalum: Min. Jour., vol. 168, No. 4927, London, Jan. 25, 1930, p.10.

114/ Wagner, P. A., Geology and Mineral Industry of Southwest Africa: Memoir No. 7, Geol. Survey of the Union of South Africa, Pretoria, 1916, p. 41.

115/ Pisar, Charles J., (American consul, Cape Town), Mineral Resources and Industry in the Mandate Territory of Southwest Africa: Consular Rept., Sept. 18, 1922, Consular File No. 4849.

116/ Reunig, E., Pegmatite und Pegmatitmineralien in Südwestafrika. Ztschr. Krist., vol. 58, Leipzig, 1923, pp. 448-459.

figures are as follows:

Year	Pounds	Value
1923	$5,600	$3,031
1925	5,022	1,347
1926	350	149
1927	15,119	20,012
1928	-	-
1929	15,250	19,418

Export statistics are not available, but producers have reported shipments to Europe in various years.

CONSUMPTION

Without adequate import and export figures, it is difficult even to suggest probabilities for the future tantalum market or to interpret the past at all accurately. It is known that most of the domestic production has been shipped to England and Germany and that quantities of Australian ore, which is of a higher grade than the American ore, are shipped to the United States. The production table (p. 14) shows that in 1922 there was a definite increase in home production and that in 1928 production was 34 times that of 1927. It was in 1922 that the manufacture of electrolytic rectifiers (in which tantalum was used) began, and it was in 1927 that the Bishop Co., at Malvern, Pa., began making use of tantalum in laboratory equipment; but how much either industry affected the increase in production is not definitely known.

PRICES

In writing of the South Dakota ores, Connolly and O'Harra reported as follows:[117]

> Previous to 1928, columbite was bought for its tantalum-oxide content alone, quotations being based on ore or concentrates containing 60 per cent tantalum oxide (Ta_2O_5). During part of 1928 purchasers paid for the columbium oxide as well as the tantalum, prices being based on ore or concentrates containing 65 per cent combined columbium and tantalum oxides (Cb_2O_5 and Ta_2O_5) For ore of this grade prices during the summer of 1928 were as high as $30 a unit, which is equivalent to $1.50 a pound. On December 1, 1928, prices were down to $13 a unit (65 cents a pound) for ore containing 60 per cent tantalum oxide, nothing being paid for the columbium content.

The following table indicates prices, in 1928 and 1929, for ore, bar, and sheet, and powder.

Domestic Prices for 1928 and 1929[1]

Product	1928	1929
Tantalum ore (per unit of Ta_2O_5, basis 60 per cent ore)...	$13 to $30	$13
Tantalum bar and heavy sheet (per gram)	20¢	16¢ to 20¢
Powder (per gram)	11¢ to 12¢	11¢

[1] Eng. and Min. Jour.

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[117] Connolly, Joseph P., and O'Harra, Cleophas C., The Mineral Wealth of the Black Hills: S Dak., School of Mines, Bull. 16, May, 1929, pp. 255-257.

COLUMBIUM

Columbium has as yet found little or no industrial application. The properties of columbium resemble very closely those of tantalum. It is inert to nearly all chemical action and is soluble in a mixture of nitric and hydrofluoric acids. "In electrolytes, the metal is unidirectional. It readily absorbs gases by occlusion and has been patented as a 'getter' in vacuum tubes. It may be rolled, drawn, hammered, formed, or cut with ordinary working tools. It welds readily to itself and other metals by the spot-welding process. However, the metal must not be subjected to any great amount of heat in the atmosphere, or it will become brittle."[118] Nevertheless, columbium has received comparatively little attention, perhaps because of the difficulties in the methods available for its extraction. The adoption of a satisfactory method of separation would not only bring into use one more of the rarer metals but would bring about a cheaper production of tantalum.

Except for about one-half ounce of metallic columbium made by Siemens in 1906, the metallic columbium exhibited recently by the Fansteel Products Co. (twelfth exposition of chemical industries) represents the world's supply, consisting of 25 pounds of the metal, in sheets, bars, rods, and wire.[119]

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[118] Engineering and Mining Journal, Columbium Now a Commercial Metal: vol. 128, No. 16, Oct. 12, 1929, p. 580.

[119] Engineering and Mining Journal, Work cited.

POSSIBLE PRODUCERS OF OR DEALERS IN TANTALUM-COLUMBIUM MINERALS

Bakelite Corporation, 247 Park Ave., New York, N. Y.
Black Hills Tin Co., Tinton, S. Dak., 1/
Collingwood and Green, Pringle, S. Dak., 1/
Consolidated Feldspar Co. (including the Keystone Feldspar & Chemical Co., Keystone, S. Dak.), Trenton, N. J., 1/
Cummins, J. W., Feldspar Co., Bath, Maine. 2/
Dakota Feldspar Co. (A. T. Roos), Rapid City, S. Dak. 1/
Endicott, James D., 326 Rudd St., Canon City, Colo.
Evans, Charles, Tip Mountain Mine, Custer, S. Dak.
Farnam, F. D., & Co., 359 N. Wells St., Chicago, Ill.
Foote Mineral Co. (Inc.), 1609 Summer St., Philadelphia, Pa.
General Minerals Corporation, Suite 317-319, Mather Bldg., Washington, D. C.
Grant, George B., Box 417, Custer, S. Dak.
Green, L. C. Pringle, S. Dak.
Hamm, B. C., Eastern Mining Co., 8A Forest St., Cambridge, Mass.
Henault, Denis, Keystone, S. Dak.
Hoyt Mineral Corporation, Las Tablas, N. Mex. 2/
Kamman, C. H., Hill City, S. Dak.
Lemmon, W. P., 106 Spring St., Johnson City, Tenn.
Ludeke, W., 97-101 Warren St., New York, N. Y.
McGowan, Patrick J., Keystone, S. Dak.
Mackay, A. D., 26 Cortlandt St., New York, N. Y.
Peerless Consolidated Copper Co., Hillside, Custer County, Colo.
Pegmatite Minerals Co., 639 F. St., N. W., Washington, D. C.
Scott, Oscar C., Searchlight, Nev.
Varlacoid Chemical Co., 15 Moore St., New York, N. Y. 2/
Wallace, W. K. 530 Fifth Ave., New York, N. Y. 2/
Werdelin, John, 1120 Fifth St., Denver, Colo.

1/ Reported production to the Bureau of Mines in 1928 (reports for 1929 not yet received).

2/ Indicated upon replies to a questionnaire sent out by the Bureau of Mines that it either produced or intended to produce ores.

BUYERS OF TANTALUM-COLUMBIUM MINERALS

Domestic

Bishop & Co., Malvern, Pa.,
Fansteel Products Co., North Chicago, Ill.
Harris Engineering Co., Los Angeles, Calif.
Metal & Ore Corporation, Woolworth Bldg., New York, N. Y.
Minerals & Metals Corporation, Merchants National Bank Bldg., Los Angeles, Calif.
Molybdenum Corporation of America, Empire Bldg., Pittsburgh, Pa.
Pfaltz & Bauer (Inc.), 300 Pearl St., New York, N. Y.
Philipp Bros. (Inc.), Woolworth Bldg., New York, N. Y.
Varlacoid Chemicals Co., 15 Moore St., New York, N. Y.

Foreign

George G. Blackwell Sons & Co. (Ltd.), The Albany, P. O. Box 22, Liverpool, England. (Purchaser of the ore.)1/
Guthrie & Co. (Ltd.), Accrington, Lancashire, England. (Manufacturer of apparatus in ductile tantalum.)
Hermann Essing & Co., Kleine Weinbergstrasse 2, Wiesbaden, Germany.
Siemens & Halske A-G, Charlottenburg, Germany. (Manufacturer of apparatus in ductile tantalum.)

1/ According to the Industrial Australian and Mining Standard, this company is the only large consumer in England of tantalite ore, which it uses for the manufacture of ferrotantalum-niobium alloy. It has purchased from Western Australia between 50 and 100 tons of ore, having approximately 80 per cent of tantalic acid; it has purchased important quantities from other countries far removed from Australia and has regular shipments coming forward at prices varying from £200 to £320 per ton. The company desires ore reasonably free from tin and asks for a statement of the approximate percentage of niobic acid in the ore, if any exists. The basis for buying is the analysis made by Dr. George Tate, of Liverpool, an expert analyst of tantalum ore.

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Andrieux, L. Current articles worth noting; the preparation and properties of the borides of tantalum and columbium. Compt. rend., Dec. 30, 1929, pp. 1279-1281. (In French.)

Armstrong, Percy A. E. Iron Alloy. U. S. Patent 1,687,486, Oct. 16, 1928. Chem. Abs., vol. 23, No. 1, Jan. 10, 1929, p. 88.

Concerns an alloy resistant to scaling at elevated temperatures.

________. Ferrous alloys containing chromium and tantalum. U. S. Patent 1,711,484, May 7, 1929. Chem. Abs., vol. 23, No. 13, July 10, 1929, p.3204

Concerns a rust, heat, and acid resisting alloy, suitable for cutlery, valves, etc.

Austin, Miner M., and Balke, Clarence W. (To Fansteel Products Co.) Apparatus for removing carbon and gases from tantalum wire. U. S. Patent 1,688,481, Oct. 23, 1928. Chem. Abs., vol. 23, No. 1, Jan. 10, 1929, P. 89.

Balke, Clarence W., Metals of the tungsten and tantalum groups. Ind. and Eng. Chem., vol. 21, No. 11, Nov., 1929, pp. 1002-1007.

Bruderus, Stahlwerke Rochling, and Kropf, Alfred. Refractory material. German Patent 469,433, Dec. 19, 1926. Chem. Abs., vol. 23, No. 7, April 10, 1929, p. 1732.

States that tantalum powder, which may contain up to 10 per cent impurity, is mixed with graphite in the proportions 3.2 with the use of a binder, e.g pitch or molasses. The mixture is molded under pressure, dried at 300-500° and heated at 2000-3000°. The molded piece may be given a coating of graphite before heating. The products may be used as electroresistance heaters or for making crucibles, furnace linings, etc.

Chemical Age (London). Some inventions of the month; electrolytic extraction of metals. Vol. 21, No. 545, Dec. 7, 1929, p. 43.

Drouot, H. Recent progress in metallurgy. Tech. moderne, vol. 20, 1928, pp. 857-861. Chem. Abs., vol. 23, No. 5, March 10, 1929, p. 1086.

Compares high and low frequency induction furnaces, describes rolling mills of great capacity, states that the properties of non-ferrous metals are greatly altered when extreme purity is attained, and discusses the properties of Be and Ta and their possible uses.

Grosse, Aristid D. The analytical chemistry of element 91, ekatantalum and its difference from tantalum. Jour. Am. Chem. Soc., vol. 52, No. 5, May, 1930, pp. 1742-1747.

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Gives data on the linear thermal expansion of three samples of worked and annealed tantalum over various temperature ranges between -190 and 500° C. A summary of available data by previous observers on the thermal expansion of tantalum is included.

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Remy-Stahlwerke Stahl Schmidt & Co. Ges. Alloy Steel for "high-speed" tools. British Patent 289,990, Feb. 28, 1927. Chem. Abs., vol. 23, No. 4, Feb. 20, 1929, p. 813.

Specifies an alloy steel containing 17 per cent W, 5 per cent Cr, 1.5 per cent V, 7 per cent Ta, and 0.9 per cent C, with or without 2 per cent Mo.

Schoeller, W. R., and Jahn, C. A new method for the separation of small quantities of tantalum and niobium from titanium. Analyst, Cambridge, England, vol. 54, 1929, pp. 320-326.

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